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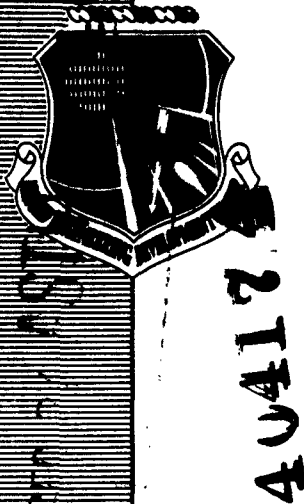
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63-3-4

CRYOPUMPING OF 77°K NITROGEN AND ARGON ON 10-25°K SURFACES

By

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Aerospace Environmental Facility

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NOTICES

ABSTRACT

Results are presented on the first phase of an experimental program designed to study the effects of gas and cryosurface temperatures on the capture coefficient in cryopumping. Experimental pumping speeds and capture coefficients were obtained for 77°K nitrogen and argon on a spherical cryosurface with temperatures ranging from 10 to 25°K. The capture coefficients for the two gases were found to follow exponential functions of the cryosurface temperature. These functions were found to be similar to the Boltzmann distribution law.

PUBLICATION REVIEW

This report has been reviewed and publication is approved.

H. K. Doetsch
Technical Advisor
DCS/Research

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ILLUSTRATIONS

NOMENCLATURE

A_c Cryosurface area, cm^2

A_j Peak height at mass j, scale divisions

a_{ij} Sensitivity factor of the mass spectrometer to the "i"th gas at mass j, scale divisions/mm Hg

C Capture coefficient

$\dfrac{dV}{dt}$ Rate of flow, liters/sec

$\left(\dfrac{dV}{dt}\right)_o$ Inflow rate of condensable gas at a pressure P_o, liters/sec

K Boltzmann constant, ergs/°K

M Molecular weight, grams/mole

n/n_o Fraction of molecules having an energy greater than ϵ_j

P Pressure, mm Hg

P_c Pressure in the chamber, mm Hg

P_o Fore pressure on the standard leak, mm Hg

P_s Vapor pressure of experimental gas, mm Hg

R Universal gas constant, ergs/mole-°K

S Pumping speed, liters/cm^2-sec

T Temperature, °K

T_g Gas temperature, °K

T_o Temperature of standard leaks, °K

T_s Cryosurface temperature, °K

W Work required to remove a molecule from the cryosurface, calories

α Proportionality constant

ΔE Molecular energy of sublimation, calories

ΔH Molecular heat of sublimation, calories

ΔV Volume change, liters

ϵ_j Energy required for a molecule to escape from the cryosurface, ergs

SUBSCRIPTS

a	Analyzer conditions
c	Chamber conditions
i	Pertaining to the "i"th species
m	Measured conditions
x	Pertaining to volume x
y	Pertaining to volume y

1.0 INTRODUCTION

The pumping speed of a cryosurface under free-molecule conditions can be predicted by kinetic theory if the proper capture coefficient is known. The capture coefficient, as defined by the ratio of the actual pumping speed to the theoretical maximum pumping speed (Ref. 1), is a measure of the fraction of molecules that stick or condense on the first collision with the cryosurface. The capture coefficient is presumably related to the energy of the gas molecules being pumped as well as the cryosurface temperature. If this relationship were known, the pumping speeds of any gas could be readily predicted. This report covers the first of a series of experiments designed to study this relationship.

With the advent of space simulation testing (Ref. 2), an accurate prediction of the system conditions during a particular test phase becomes a necessity. One of the important test parameters is the pressure. In order to predict the system pressure, the gases present as well as their cryopumping speeds or capture coefficients must be known. Presumably, the composition will be known; however, the capture coefficients are not generally available.

Since the operating cryosurface temperatures of most space simulation chambers have been set at 20°K, the range of cryosurface temperatures chosen for these experiments includes this temperature. Experimental pumping speeds and capture coefficients are presented for nitrogen and argon over a cryosurface temperature range of 10 to 25°K. A preliminary theoretical treatment of the data is also presented.

2.0 APPARATUS

The apparatus used in this study is shown in Fig. 1. It consists essentially of a vacuum chamber containing a spherical cryosurface and an independent gas addition system. The gas addition system (Fig. 2) consists of a surge tank, a barometer for measuring the upstream gas pressure, and a series of five calibrated standard leaks arranged in parallel, thus allowing individual use of each leak or any combination of leaks. Individual valves accompany each standard leak, and the complete gas addition system has a separate pumping station.

Manuscript received February 1963.

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The vacuum chamber is a 20 x 39-in. stainless steel cylinder of all heliarc-welded construction. The chamber is suspended in an insulated stainless steel tank which was filled with liquid nitrogen during the experiments. After correction for the pump lines and cryosphere, the chamber volume was calculated to be 202 liters. This chamber size enables one to use commercially available standard leaks, without introducing significant errors in the time measurement when calibrating the gas analyzer by the rate of pressure rise method. The 7-in.-diam cryosphere has a surface area of 995 cm^2. In order to prevent condensation on the cryosphere transfer lines inside the vacuum chamber, a vacuum jacket is employed. Because of thermal conduction, a cryodeposit does form a short distance on the vacuum-jacketed line from the cryosphere. However, this excess area compensates for the area on the cryosphere covered by the transfer line. Thus, no area correction is required.

The vacuum chamber pumping system consists of a 6-in. oil diffusion pump with both liquid nitrogen and water-cooled baffles. The diffusion pump is backed by a small mechanical roughing pump. The chamber can be isolated from the pumping system by a 2-in.-high vacuum gate valve.

The cryosphere was cooled to temperatures ranging from 10 to 25°K by a helium cryostat converted to a refrigeration cycle. The temperature of the cryosphere was controlled by a bypass valve on the cryostat, which simply diverted part of the helium flow from the cryosphere, thus, reducing its cooling capacity. The temperature of the cryosphere was measured by a hydrogen vapor pressure thermometer located inside the cryosphere. The temperature drop through the metal wall of the sphere was calculated and found to be negligible.

A residual gas analyzer was the primary instrumentation in the experiments. An ionization gage was mounted on the pump-out line next to the vacuum chamber to monitor the turning on and off of the analyzer and diffusion pump.

The entire vacuum system was equipped with outgassing heaters capable of producing temperatures up to 500°F.

3.0 PROCEDURE

With the vacuum chamber at operating conditions, that is, with the chamber immersed in liquid nitrogen, the cryosphere at the desired temperature (10 - 25°K), and the chamber pressure below 5×10^{-8} mm Hg, the following experimental technique was employed. First, the cryosphere

was coated with the experimental gas (N_2 of A) by introducing the gas through the 10^{-1} cc/sec standard leak for 30 minutes. The purpose of this preliminary coating was to eliminate the "bare surface" effect (Ref. 1). After precoating and with the chamber back to operating conditions, the experimental gas was introduced at a constant rate into the chamber and the system allowed to reach a steady-state condition. The constant inleakage rate was accomplished by flowing the gas through a calibrated standard leak and maintaining the back pressure at a known constant value. When a steady state was reached, as determined from the gas analyzer, the partial pressures of each gas present were determined by scanning the mass spectrum with the gas analyzer. The flow of gas into the chamber was then stopped by closing a valve in the gas line at the entrance to the chamber, and the system was allowed to reach equilibrium. The partial pressure of the experimental gas at these new conditions was determined by the same method mentioned previously. The pressure drop caused by cryopumping the experimental gas was then the difference between the two partial pressures. The difference in partial pressures were taken due to the background of mass 28 in the mass spectrometer. Carbon monoxide also gives a peak at mass 28 and is indistinguishable from N_2 when present in small quantities. This correction was less than 1.5 percent for the N_2 runs and there was no detectable background for argon, therefore no correction was made. From the measured data, the pumping speed of the cryosphere could be determined.

This procedure was followed over a range of cryosurface temperatures and chamber pressures ranging from 10^{-8} mm Hg to 10^{-5} mm Hg.

4.0 CALIBRATION OF THE RESIDUAL GAS ANALYZER

The residual gas analyzer used in the experiments is a 60-deg sector field instrument of the Nier design. The analyzer uses one permanent magnet with a soft iron shunting bar to yield two fixed magnetic fields for high or low mass scans. It is possible to scan a mass range of 44-2 with unit resolution. The instrument will detect partial pressures of 5×10^{-12} mm Hg; however, the output is linear with pressure only over a pressure range of 1×10^{-10} mm Hg to 1×10^{-4} mm Hg. That is,

$$a_{ij} P_i = A_j \quad \text{for} \quad 10^{-10} < P_i < 10^{-4} \tag{1}$$

where P_i is the partial pressure of the "i"th gas in mm Hg, A_j is the peak height (recorded output from the analyzer) at mass j in scale divisions, and a_{ij}, measured in units of scale divisions per mm Hg, is the proportionality constant or sensitivity factor of the instrument to the "i"th gas at mass j.

The analyzer was calibrated for each gas studied in this report. The calibration technique used was to introduce a known amount of gas i into the system, measure the change in peak height, A_j, and then calculate a_{ij} from Eq. (1). The sensitivity factor was determined under both static and dynamic conditions and corrected for the thermal transpiration effect, which will be discussed at length in the next section. The values of a_{ij} for nitrogen and argon thus determined are 38.5×10^8 and 43.5×10^8, respectively. The total variance in a_{ij} in eight such calibrations for each gas was less than two percent.

4.1 THERMAL TRANSPIRATION EFFECT

When two volumes are separated by a small orifice or porous plug such that the mean free path is large compared to the opening and steady-state conditions exist, the pressure in volume x is to the pressure in volume y as the square root of the ratio of their temperatures (Ref. 3). That is,

$$\frac{P_x}{P_y} = \sqrt{\frac{T_x}{T_y}} \qquad (2)$$

The following diagram illustrates the vacuum chamber and the location of the gas analyzer.

Analyzer at
Room Temperature

Vacuum
Chamber
77°K
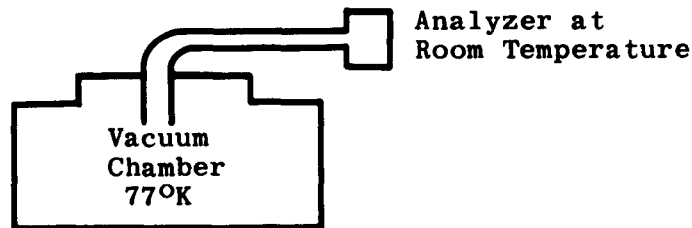
The tube connecting the analyzer to the chamber is approximately 15 in. long and has a 0.6-in. inside diameter. Since the gas in the chamber is at 77°K, the gas in the analyzer is at room temperature (300°K), and free-molecule flow exists with respect to the tube opening, a thermal transpiration definitely exists between the chamber and the analyzer. Since the pressure is measured at the analyzer and the desired pressure is that in the chamber, the thermal transpiration effect must be considered. That is,

$$P_c = P_a \sqrt{\frac{T_c}{T_a}} \qquad (3)$$

where subscript a refers to conditions at the analyzer and subscript c refers to conditions in the vacuum chamber.

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In order to explain this fact as it pertains to the specific system in this report, it is necessary to examine the pumping speed equation.

$$S = \frac{P_o}{P_c}\left(\frac{dV}{dt}\right)_o \frac{1}{A_c} \tag{4}$$

This equation gives the pumping speed of the cryosurface when the entire system is at a constant temperature.

S is the pumping speed in liters/cm²-sec

P_o is the fore pressure on the standard leak in mm Hg

P_c is the steady-state chamber pressure in mm Hg

$\left(\frac{dV}{dt}\right)_o$ is the volume flow rate through the standard leak at P_o, T_o in liters/sec

A_c is the surface area of the cryosphere in cm²

In order to have the equation in terms of the measured quantities, it is necessary to consider the temperature effects.

The flow rate is determined by the equality

$$\frac{P_o}{T_o}\left(\frac{dV}{dt}\right)_o = \frac{P_c}{T_c}\left(\frac{dV}{dt}\right)_c \tag{5}$$

or

$$P_c\left(\frac{dV}{dt}\right)_c = \frac{P_o T_c}{T_o}\left(\frac{dV}{dt}\right)_o \tag{6}$$

where subscript c refers to chamber conditions and subscript o refers to the measured conditions.

For this particular system,

$$T_o = T_a = 300°K$$

and

$$T_c = 77°K$$

Therefore,

$$S = \frac{\frac{P_o T_c}{T_o}\left(\frac{dV}{dt}\right)\frac{1}{A_c}}{P_a \sqrt{\frac{T_c}{T_a}}}$$

$$= \frac{P_o}{P_a}\sqrt{\frac{77}{300}}\left(\frac{dV}{dt}\right)_o \frac{1}{A_c}$$

$$= 0.51 \frac{P_o}{P_a}\left(\frac{dV}{dt}\right)_o \frac{1}{A_c} \tag{7}$$

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As stated previously,

$$P_a = P_i = \frac{A_j}{a_{ij}}$$

Thus,

$$S = \frac{0.51 \ P_o \ a_{ij}}{A_j} \left(\frac{dV}{dt}\right)_o \frac{1}{A_c} \qquad (8)$$

When the gas analyzer was calibrated with the chamber at room temperature, the sensitivity factor for nitrogen was determined by the method previously described to be 77×10^8 scale divisions per mm Hg. Using this factor and substituting P_i from Eq. (1) into Eq. (4), the pumping speed is given by

$$S = \frac{P_o \ (77 \ x \ 10^8)}{A_j} \left(\frac{dV}{dt}\right)_o \frac{1}{A_c} \qquad (9)$$

The analyzer was also calibrated with the chamber at 77°K. The sensitivity factor in this case was 38.5×10^8 scale divisions per mm Hg, and the equation for the pumping speed is

$$S = \frac{P_o \ (38.5 \ x \ 10^8)}{A_j} \left(\frac{dV}{dt}\right)_o \frac{1}{A_c} \qquad (10)$$

A comparison of Eqs. (9) and (10) under the same flow conditions shows a difference of a factor of 0.50. However, in Eq. (9) the measurements were made with the entire system at 300°K, whereas in Eq. (10) part of the system was at 77°K. Thus, the calibration of the analyzer under the two different conditions has, in fact, agreed with the square root of the temperature ratio in the pumping speed calculation, and a_{ij} determined with the chamber at 77°K includes the correction for thermal transpiration. The ratio of the sensitivity factors is then a measure of the thermal transpiration effect.

The calibration for argon gave a sensitivity factor of 87×10^8 scale divisions per mm Hg for a 300°K system and 43.5×10^8 scale divisions per mm Hg for a system with a 77°K chamber.

5.0 RESULTS

Cryopumping speeds were determined for 77°K nitrogen and argon over a pressure range of 10^{-8} mm Hg to 10^{-5} mm Hg. For nitrogen the pumping speed data were taken at measured cryosurface temperatures of 10, 12.5, 15, 20, 23, and 25°K. The results are shown in Fig. 3 as pumping speed vs pressure. (Because of the purity of the gases used in these studies, the partial pressure of the experimental gas is essentially

equal to the chamber pressure). For argon the pumping speed data were taken at measured cryosurface temperatures of 10, 12.5, 15, 20, and 25°K. The results are shown in Fig. 4.

From the pumping speed data, the capture coefficient at each cryosurface temperature was calculated. The capture coefficient is defined as

$$C = \frac{S}{\sqrt{RT_g/2\pi M} \left(1 - \frac{P_s}{P_c}\right)} \qquad (11)$$

where S is the experimental pumping speed, R is the universal gas constant, M is the molecular weight of the condensable gas, T_g is the temperature of the condensable gas, P_s is the vapor pressure of the condensable gas at T_s in °K, and P_c is the chamber pressure.

Over the stated experimental pressure range, the vapor pressure correction factor, $\left(1 - \frac{P_s}{P_c}\right)$, for these experiments is equal to one, since the ratio of the vapor pressure to the chamber pressure is small. The only exception to this is for nitrogen on 23 and 25°K cryosurfaces. For these data, the pressure range was limited to 10^{-6} to 10^{-5} mm Hg to ensure that the effect of the vapor pressure correction would be small with respect to experimental error. Since the chamber was at liquid nitrogen temperature and free-molecule conditions existed, the gas temperature would be that of the chamber walls, i. e., 77°K.

Thus,

$$C = \frac{S}{6.04} \quad \text{for } N_2$$

$$C = \frac{S}{5.06} \quad \text{for } A$$

Figures 5 and 6 show the capture coefficient plotted as a function of cryosurface temperature for N_2 and A, respectively.

6.0 ANALYSIS OF RESULTS

The distribution of energy among the various gas molecules in a closed system is given by the Boltzmann distribution law (Ref. 5)

$$\frac{n}{n_o} = e^{-\epsilon_j/KT_g} \qquad (12)$$

where $\frac{n}{n_o}$ is the fraction of molecules having an energy greater than ϵ_j, K is the Boltzmann constant, and T is the gas temperature. When the

gas temperature is increased, the fraction of molecules having an energy greater than e_j is increased proportionally. By analogy, when considering a system where the gas molecules are impinging on a cryosurface, one lets ϵ_j be the energy required for a molecule to escape from a cryosurface, then the fraction that rebound or escape after colliding with the surface would be $\frac{n}{n_0}$. The fraction that escape or rebound from the surface increases as the gas temperature increases, because as the gas temperature is raised the average molecular energy is increased. At a constant cryo-surface temperature, the energy required to escape from the cryosurface is constant; therefore, any increase in the average energy of the gas molecules will increase the fraction of molecules that escape from the surface. As illustrated by the experimental data in Figs. 5 and 6, the capture coefficient, C, for a gas at a constant temperature decreases with increasing cryosurface temperatures. Since the capture coefficient, C, is defined as the fraction of molecules that condense on collision, 1 - C equals the fraction that rebound or

$$1 - C = \frac{n}{n_0} \tag{13}$$

Thus, the main problem is to establish a method for calculating the escape energy, ϵ_j, for various gas molecules. As previously stated, the fraction 1 - C is a function of the gas and cryosurface temperatures as well as the internal energy of the molecule, ΔE. That is, $\frac{\epsilon_j}{KT}$ equals $f(T_g, T_s, \Delta E)$. Since ϵ_j is the energy necessary for the molecule to escape from the cryosurface, it is assumed to be proportional to the internal energy of the molecule, ΔE, which may be calculated from the thermodynamic relationship

$$\Delta E = \Delta H - P\Delta V = \Delta H - W \tag{14}$$

where ΔH is the molecular heat of sublimation at the cryosurface tem-perature, T_s, and W the work required to remove one molecule of substance from the cryosurface. In Eq. (12), ϵ_j is specified at the gas temperature, T_g, but ΔE is related to the cryosurface temperature rather than to the gas temperature. Then Eq. (12) may be expressed as

$$\frac{n}{n_0} = 1 - C = e^{-\epsilon_j/KT_g} = e^{-\frac{\beta \Delta E/KT_s}{KT_g}} = e^{-\alpha \Delta E/T_s} \tag{15}$$

where α and β are proportionality factors. Since in this study the gas temperature is held constant, it enters Eq. (15) in the factor α. Substi-tution for ΔE in Eq. (15) from Eq. (14) yields

$$1 - C = e^{[-\alpha(\Delta H - W)]/T_s} \tag{16}$$

The values of ΔH-W for nitrogen and argon are given in the following table:

T_s, °K	Gas	ΔH-W, cal/mole
10	N_2	1590
15	N_2	1580
20	N_2	1570
25	N_2	1560
10	A	1980
15	A	1970
20	A	1960
25	A	1950

The values for the heat of sublimation of nitrogen and argon were obtained from Refs. 6 and 7, respectively. The work required to remove a molecule from the cryosurface, W, equals PΔV, where ΔV is the difference in volume between one mole of the gas and one mole of solid. The volume of the solid is negligible compared to the volume of the gas; therefore, PΔV equals PV. Considering the gas to be ideal, PV equals RT or W equals RT, where T is the temperature of the cryo-surface. The constant α was calculated from Eq. (16) using the average experimental value of C from the 10°K experiments. The values thus obtained for α are 0.0324 for N_2 and 0.0455 for A. The solid lines in Figs. 5 and 6 represent the capture coefficients calculated from Eq. (16). The lower temperature limit of the capture coefficient as given by Eq. (16) is 1.0 at T_s = 0°K. The upper temperature limit, however, is governed by several factors: the most important are the vapor pressure of the condensate and the difference in temperature between the cryo-surface and the chamber walls. If one extrapolates the capture coef-ficient plot in Fig. 5 to a capture coefficient of zero, the intercept is at a cryosurface temperature of approximately 77°K. This is to be expected since if the cryosurface temperature were equal to the gas temperature, only physical adsorption would occur and equilibrium con-ditions would exist. However, it must be pointed out that the vapor pressure of the condensate will have a greater effect on the capture coef-ficient at cryosurface temperatures above 25°K, making such an extrapo-lation very dubious. This correction at 25°K is less than one percent at a chamber pressure of 1 x 10^{-5} mm Hg (Ref. 8).

Although Eq. (16) expresses the experimental data quite well, it is still empirical in nature. Before a complete theoretical expression can be derived, more information must be obtained on the capture coefficient as a function of surface and gas temperature. Also, a number of various gases must be studied which will give an indication of how the capture coefficient is influenced by excitation of the various internal modes of energy and intermolecular forces.

7.0 CONCLUSIONS

The empirical equation

$$1 - C = e^{[-a(\Delta H - w)]/T_s}$$

expresses the experimental data for 77°K nitrogen and argon, but before this expression can be extended to other gas temperatures and other species, more experimental data are required. The constant a is definitely a function of gas temperature, T_g, and also a function of molecular and intermolecular quantities yet to be determined.

The following conclusions are drawn from the experimental data obtained thus far in an investigation of the effects of gas and cryosurface temperatures on the capture coefficient in cryopumping:

1. For this particular apparatus in which the chamber gas temperature was different from the gas temperature where the pressure was measured, a thermal transpiration was shown to exist and to be equal to the square root of the temperature ratio. For other studies of this type where non-isothermal conditions exist, the possibilities of thermal transpiration effects should not be neglected.

2. The capture coefficient for 77°K nitrogen and argon varies exponentially with the cryosurface temperature from 10 to 25°K. This exponential has been shown to be similar to the Maxwell-Boltzmann law.

3. As illustrated in Figs. 5 and 6, the capture coefficient cannot be arbitrarily taken as 1.0 on a 20°K cryosurface as has been done in the past for air.

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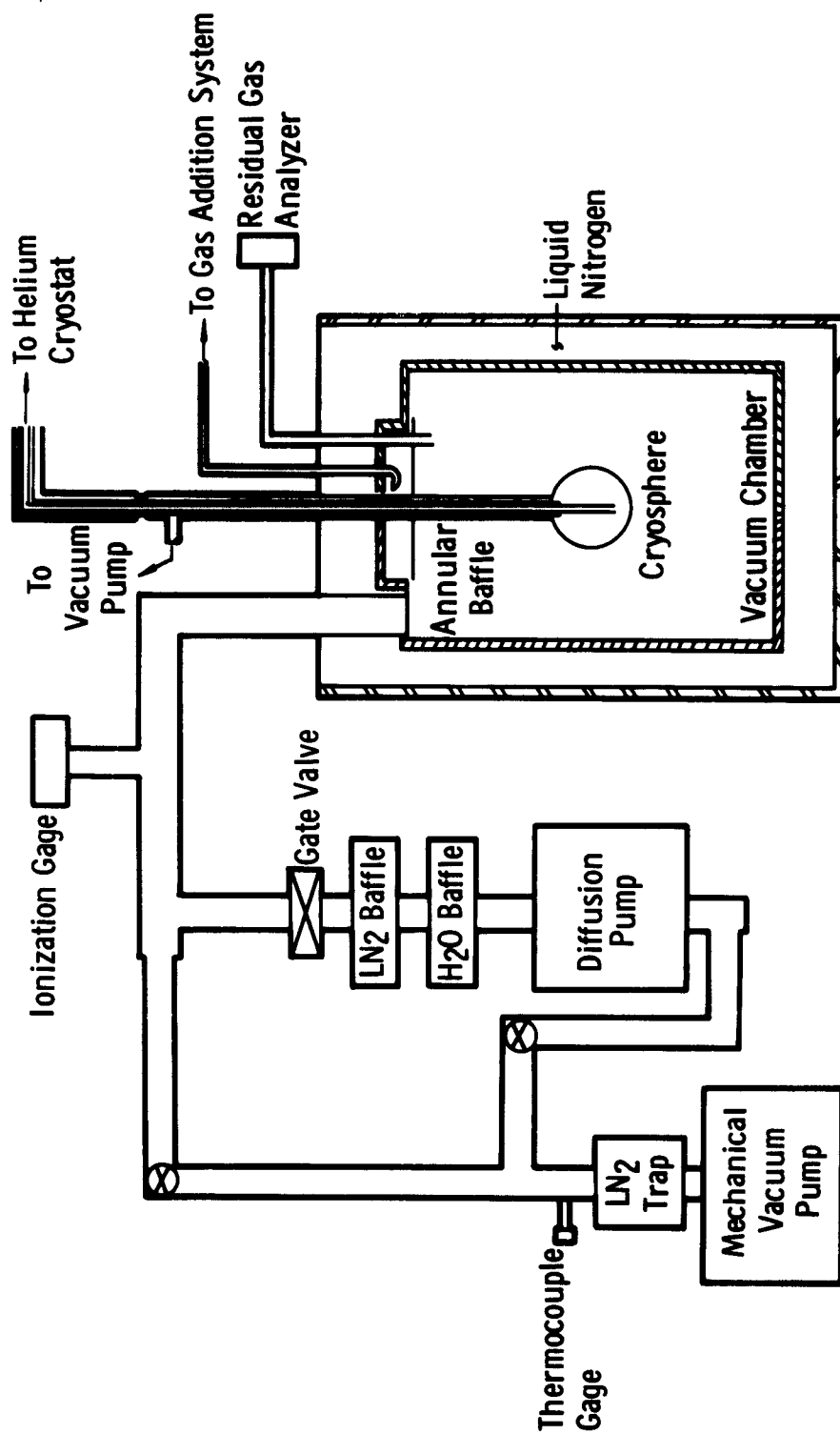
Fig. 1 Schematic Diagram of the Experimental Apparatus
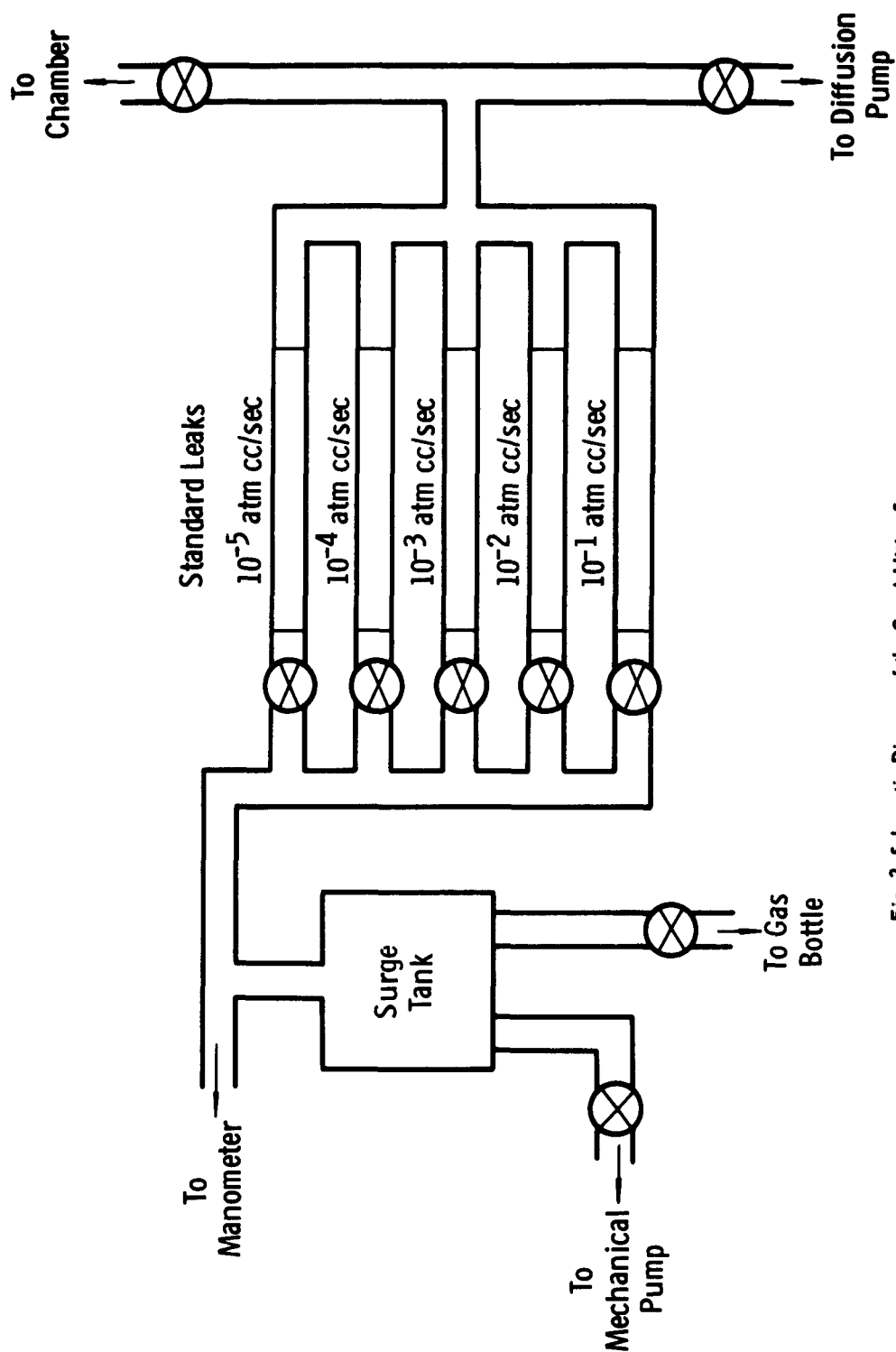
Fig. 2 Schematic Diagram of the Gas Addition System

a. 10°K Surface
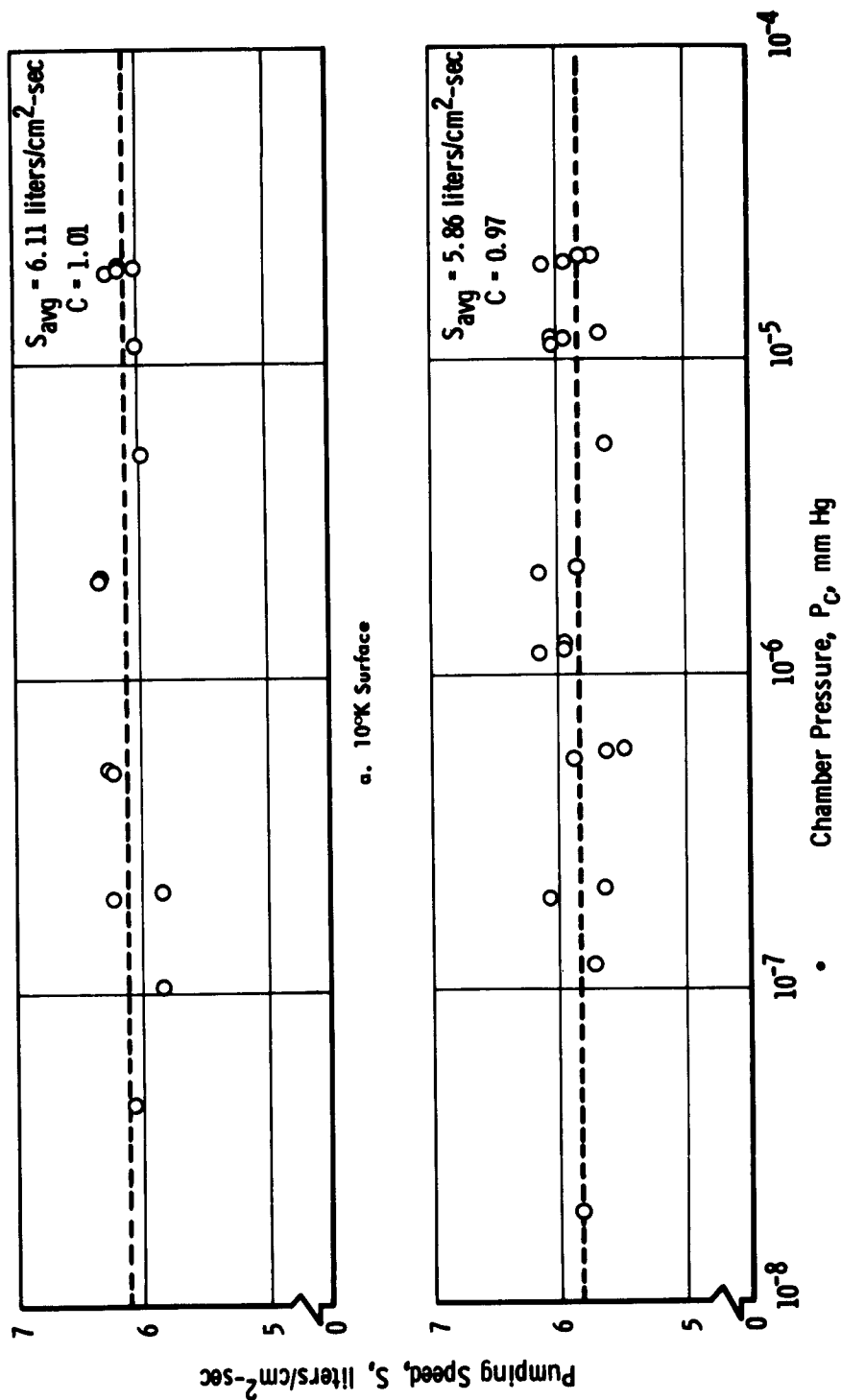
b. 12.5°K Surface

Fig. 3 Pumping Speed vs Pressure for 77°K Nitrogen

14
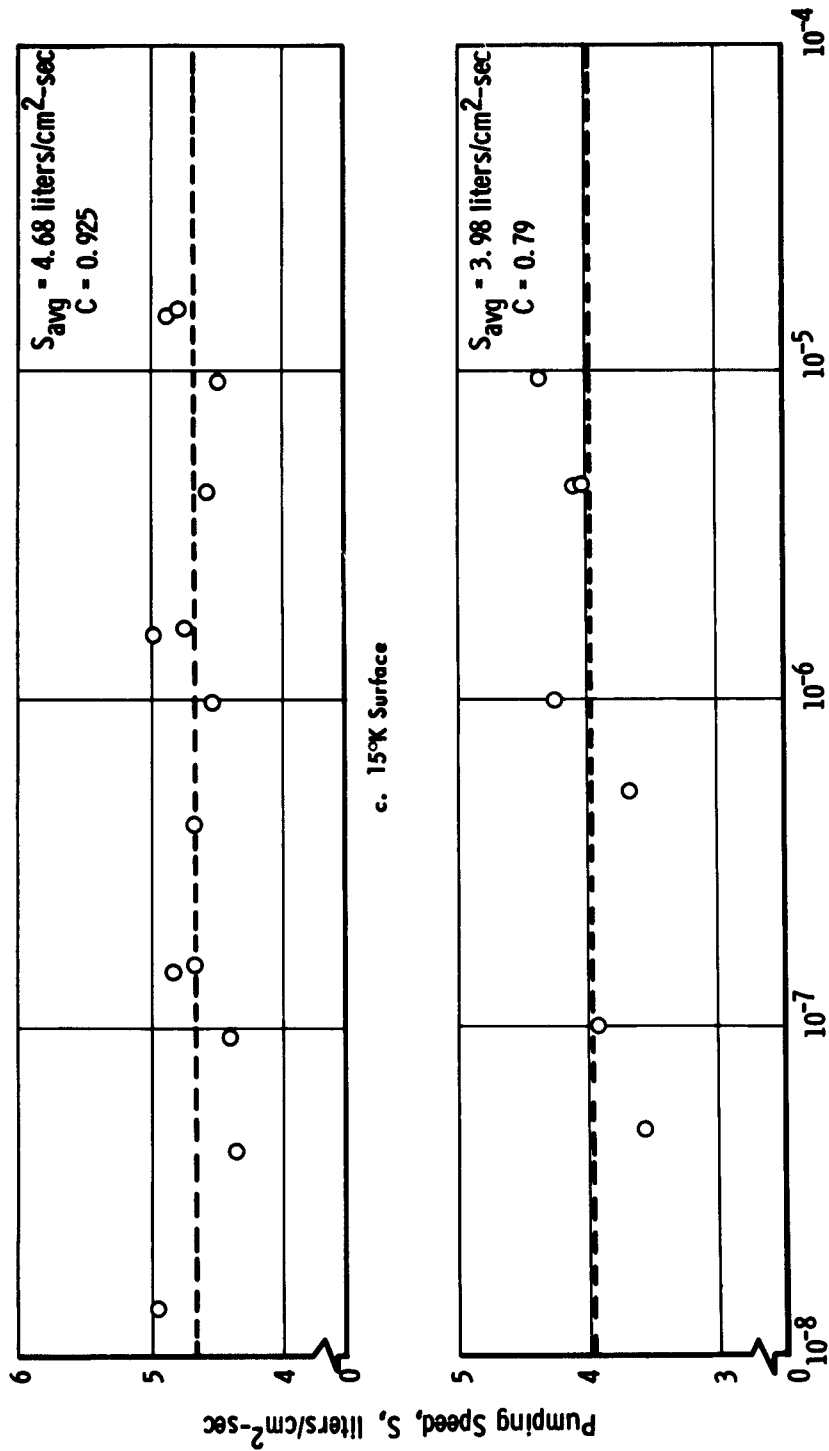
c. 15°K Surface

d. 20°K Surface

Fig. 3 Continued

e. 23°K Surface

f. 25°K Surface

Fig. 3 Concluded

Chamber Pressure, P_c, mm Hg

Pumping Speed, S, liters/cm²-sec

S_{avg} = 4.83 liters/cm²-sec
C = 0.80

S_{avg} = 4.66 liters/cm²-sec
C = 0.77

16

a. 10°K Surface

b. 12.5°K Surface

Chamber Pressure, P_c, mm Hg

Pumping Speed, S, liters/cm²-sec
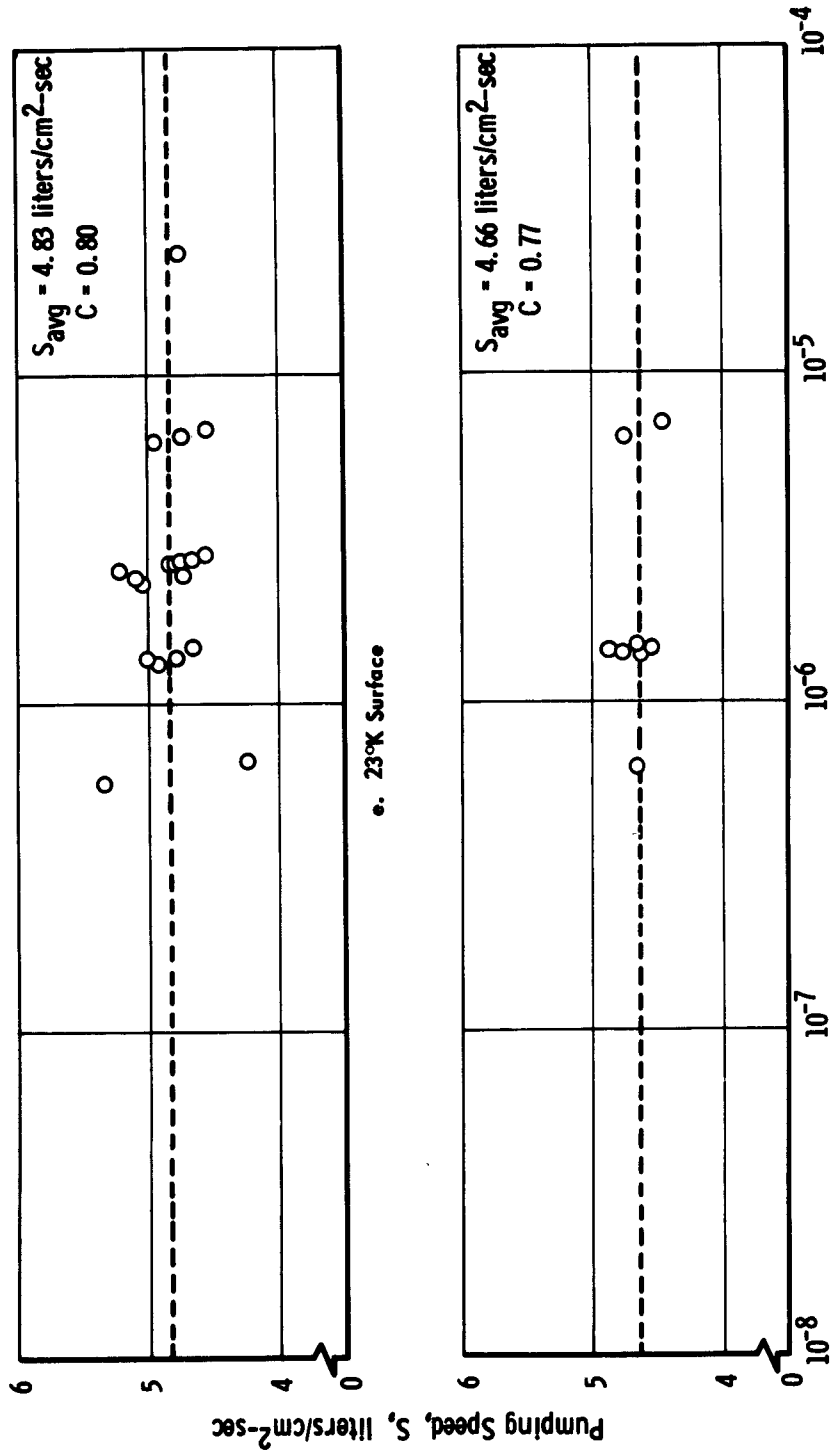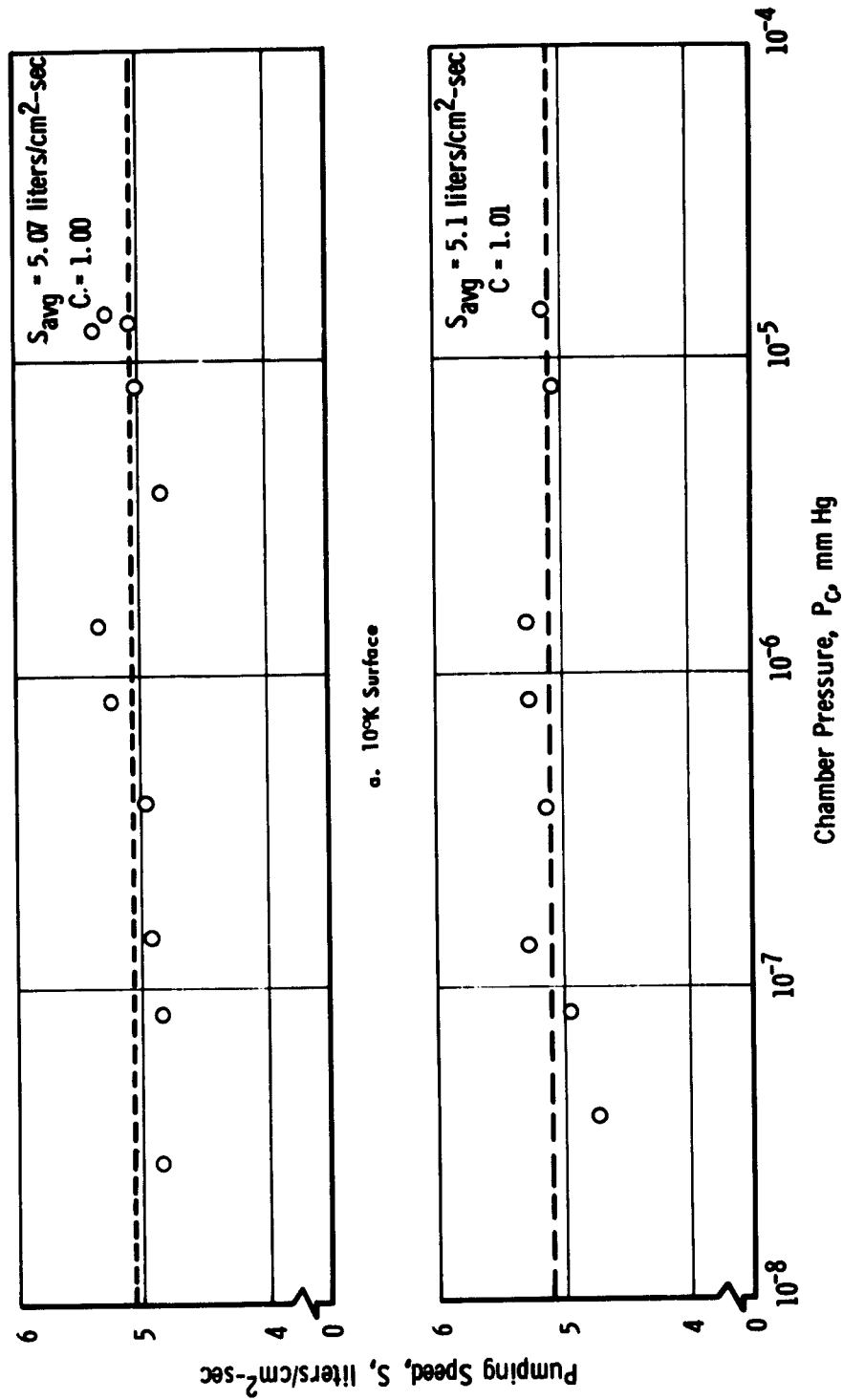
Fig. 4 Pumping Speed vs Pressure for 77°K Argon

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c. 15°K Surface

d. 20°K Surface

Fig. 4 Continued

Chamber Pressure, P_C, mm Hg

Pumping Speed, S, liters/cm^2-sec

S_{avg} = 5.98 liters/cm^2-sec
C = 0.99

S_{avg} = 5.25 liters/cm^2-sec
C = 0.87

18
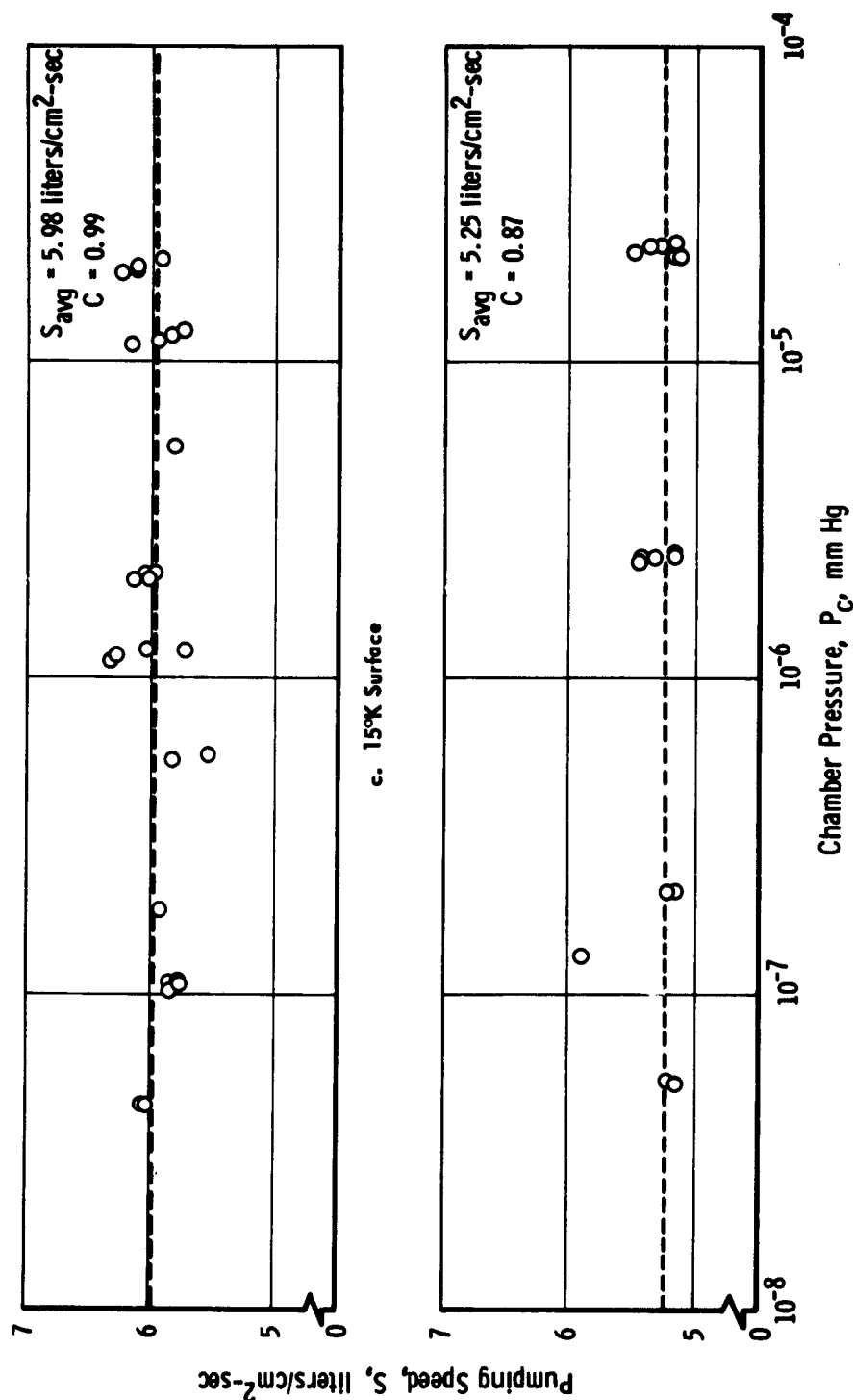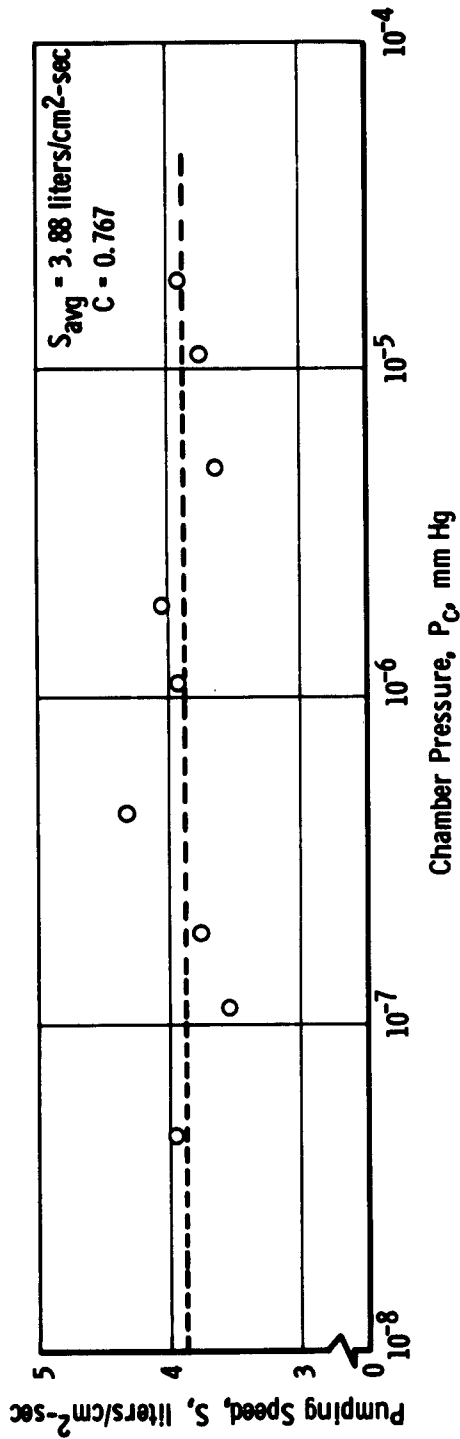
e. 25°K Surface

Fig. 4 Concluded

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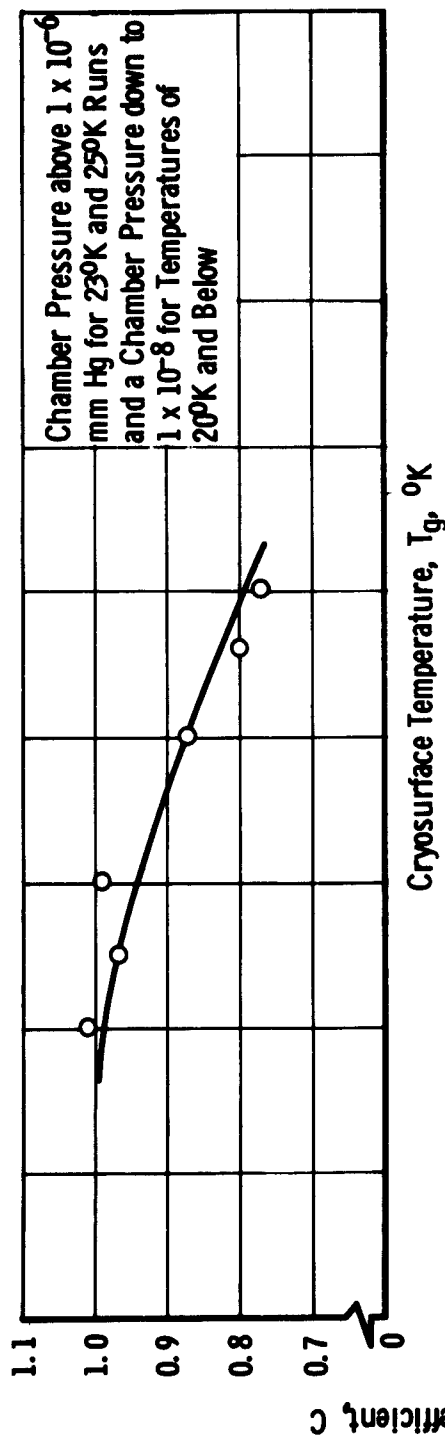
Fig. 5 Capture Coefficient vs Cryosurface Temperature for 77°K Nitrogen
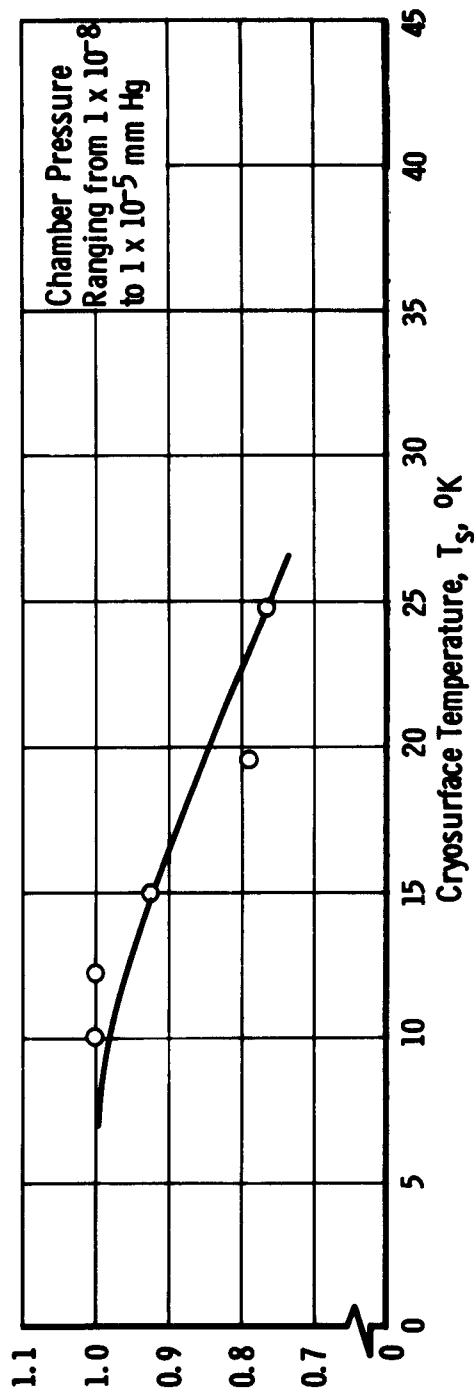
Fig. 6 Capture Coefficient vs Cryosurface Temperature for 77°K Argon

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Results are presented on the first phase of an experimental
program designed to study the effects of gas and cryo-
surface temperatures on the capture coefficient in cryo-
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spherical cryosurface with temperatures ranging from 10
to 25°K. The capture coefficients for the two gases were
found to follow exponential functions of the cryosurface
temperature. These functions were found to be propor-
tional to the Boltzmann distribution law.

1. Cryogenics
2. Surfaces
3. Vacuum pumps
4. Nitrogen
5. Argon
I. AFSC Program Area 806A,
   Project 8951, Task 89107
II. Contract AF 40(600)-1000
III. ARO, Inc., Arnold AF Sta.,
    Tenn.
IV. J. A. Collins, Jr. and
    J. P. Dawson
V. Available from OTS
VI. In ASTIA Collection